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PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements in Caulking and the like Compositions

We, ESSO RESEARCH AND ENGINEERING COMPANY, a Corporation duly organised and existing under the laws of the State of Delaware, United States of America, of Elizabeth, New Jersey, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to improvements in caulking and the like compositions.

The compositions of the invention contain a major proportion of butyl rubber or of depolymerised butyl rubber, and wax, preferably a petroleum derived wax, for example petrolatum, together with fillers and, preferably, plasticizers. The compounds of the invention may be employed as putties capable of ready working by hand without adhering to the fingers of the operator. In an alternative form of the invention the compositions may be in the form of self-sealing adhesives suitable, for example, as self-sealing adhesives for envelopes, or as sprayable compositions and, in this case, the composition will be carried in a major proportion of a solvent or mixture of solvents.

The invention, when employed for the production of putty-like materials, is illustrated by way of example in the following table (Table I).

TABLE I

Composition	Range	Parts by Weight Preferred
Enjay Butyl (065) (1)	100	100
Petroleum wax (MP 50° C.)	50—130	90
Hydrocarbon solvent (white spirit)	0—15	5
Process Oil (Light Oil)	10—50	25
Hydrated Silica (Hisil 233)	5—25	15
Soft clay (Devolite*)	50—130	85
Precipitated Whiting (Calofort* U)	0—30	10

* Devolite and Calofort are Registered Trade Marks.

(1) Mooney viscosity (large rotor) of 41 to 49 (8 minutes at 212° F.)

A typical blending procedure for the composition of Table I is as follows:—

Internal Mixer of Banbury Type

0 min.	Butyl	200° F.
1 min.	Hisil Whiting	
3 min.	½ clay ½ oil 1/3 wax	300° F.
3½ min.		Steam off, cooling water on
5 min.	½ clay ½ oil 1/3 wax	
6 min.	1/3 wax solvent	
8—9 min.	Dump	250° F.

Tack and adhesion are controlled by the ratio of wax to oil and solvent. Plasticity is also controlled by the proportions of these constituents and of filler which latter also controls the flow properties of the compositions. If depolymerised butyl rubber is employed less plasticizer is required. A depolymerised butyl rubber having a Mooney viscosity of M.L. 1+4 at 100°C. (Large Rotor) of between 20 and 70 may be suitably employed for the invention. Table II illustrates an example composition of the invention employing depolymerised butyl rubber.

TABLE II

Composition	Range	Parts by Weight Preferred
Enjay Butyl 065	100	100
Hard clay (Spestone)	40—200	150
Petroleum wax (M.P. 50° C.)	30—90	50
Ground Whiting	60—150	100
Solvent (white spirit)	0—20	5
Process oil (Light Oil)	0—25	10
Hydrated silica	5—25	10

A suitable blending procedure for the composition of Table II is the following:—

Internal Mixer of Banbury Type — Stage I — depolymerisation of butyl by action of hard clay in mixer

0 min.	Butyl	200° F.
½ min.	1/3 clay	
1 min.	1/3 clay	
1½ min.	1/3 clay	220° F.
2 min.		Steam off, cooling water on
10 min.	Dump	200° F.

If necessary to further depolymerise, cool, recharge to Banbury mixer and mix for 8 minutes finishing at 250° F. and dump.

Stage II

0 min.	Depolymerised Butyl and hard clay	200° F.
1 min.	Hydrated silica Whiting	
3 min.	½ oil 1/3 wax	
3½ min.		Steam off, cooling water on
5 min.	1/3 wax ½ oil	
6 min.	1/3 wax solvent	
8—9 mins.	Dump	250° F.

The putty-like compositions of the invention have good adhesion to a wide variety of materials including glass, metals, stoneware, and wood, have good ageing properties and do not readily flow, harden or crack. They also have other uses such as cleansers, for example by pressing pieces of the putty on to the surface to be cleaned, e.g. typewriter keys, and then removing it with the dirty material adhering to the putty. Furthermore, by the addition of suitable pigments, they can be produced in a range of colours. They are comparatively cheap particularly since slack wax can be used as the wax component. The employment of slack wax enables a high viscosity butyl rubber (e.g. $M.L._{1+4}$ at 100°C. of more than 70) to be used and this, in turn, permits a higher proportion of extending and filling materials to be employed. Typical formulations, incorporating slack wax, are shown in Table III.

TABLE III

(All parts are by weight)

Composition	1	2	3	4	5
Enjay Butyl 268 (2)	100	100	100	100	100
Ground Whiting	200	200	400	400	2000
Petrolatum (slack wax)	100	300	100	300	300

Characteristics: 1: very rubbery; 2: very rubbery and sticky;

3: rubbery; 4: rubbery and sticky;

5: 'Dry' and easily worked.

(2) Mooney viscosity (large rotor) of 50 to 60 (3 minutes at 260° F.)

A strip made from composition 5 was hung in an oven at 90°C. After 8 days it showed no signs of flow and appeared to be still in its original state.

By reducing the amount of wax and increasing the proportion of solvent, the compositions of the invention may be produced in the form of adhesives having self-sealing properties. Table IV examplifies a composition of this nature.

TABLE IV

(All parts are by weight)

Constituent		Range	Amount by Weight Preferred
Esso Butyl 365 (3)		100	100
(4)	Paraffin Wax (M.P. 50°C.)	35—65	24
	Okerin 857 (a micro-crystalline wax)		24
(5)	Devolite clay	40—80	24
	Whiting		40

(3) Mooney viscosity (large rotor) 41 to 49 (8 minutes at 212° F.)

(4) The waxes are employed, preferably, in equal amounts.

(5) Clay and Whiting are employed, preferably, in the ratio of 1 part clay of to 2 parts of Whiting.

The foregoing composition, when diluted to about 25% solids content in a 50/50 mixture of toluene and hexane, may be spread, for example on paper, and dries quickly at room temperature yielding a non-tacky film that adheres well to the surface upon which it is spread. The film is self-adhesive.

The compositions of the invention may also be prepared in the form of sprayable protective coatings, and Table V illustrates a typical compound of this type.

TABLE V

Composition	Parts by Weight
Butyl rubber	100
Wax	10—60
Silica	5—20
Clay	5—20

5—20% of the foregoing is dissolved in a suitable solvent, e.g. 50% white spirit, 50% toluene.

Sprayable compositions, as above, are useful, for example, as temporary protective finishes for the exterior trim of car bodies where damage can be considerable giving rise to reject rates of as much as 30%. The sprayable compositions of the invention can be applied at a fast rate, are fast drying and can be based on solvents, such as that indicated in Table V, that do not attack the body finish. They can be sprayed on in sufficient thickness to give adequate protection but do not adhere so firmly to the car body as to prevent them from being easily stripped off. Any residual wax adhering to the car body will be an aid to initial polishing.

WHAT WE CLAIM IS:—

1. A composition comprising a blend of a butyl rubber, a petroleum derived wax and a mineral filler.

2. A composition as claimed in claim 1 wherein the butyl rubber is depolymerised butyl rubber.

3. A composition according to any of the preceding claims containing a plasticizer.

4. A composition according to any of the preceding claims containing a solvent or mixture of solvents.

5. A composition according to claim 5 wherein the solvent is at least 75% by weight of the composition.

6. A composition according to any of the preceding claims wherein the said wax is petrolatum.

7. A composition as claimed in claim 1 and substantially as described herein.

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